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VERIFICATION OF TRANSLATION

In the Matter of Application for
Letters Patent by Matsubayashi et al.
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[Designation of Document] SPECIFICATION

[Title of the Invention] PRESS-FORMED CAN USING A PRE-COATED
STEEL PLATE

[Claims]

[Claim 1] A can having excellent corrosion resistance formed by press-forming a pre-coated steel plate which is obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer of an alloy of iron and at least one metal selected from tin, zinc and nickel, containing tin in an amount in a range of not smaller than 0.05 g/m^2 but smaller than 1.5 g/m^2 in the case of a tin-containing alloy, or containing zinc or nickel in an amount in a range of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 in the case of a zinc- or nickel-containing alloy, a silane coupling agent-treated layer containing Si in an amount of 0.8 to 18 mg/m^2 , and a thermoplastic polyester resin layer having a thickness of 8 to $42 \text{ }\mu\text{m}$ in this order from the side of the steel plate.

[Claim 2] A can according to claim 1, wherein said silane coupling agent-treated layer is a layer formed by the treatment by using an amino group-containing silane solution and/or an epoxy group-containing silane coupling agent solution.

[Claim 3] A can according to claim 1, wherein said silane coupling agent-treated layer is a layer formed by the treatment by using a mixed solution of a silane coupling agent containing an amino group and/or an epoxy group and a silane containing an organic substituent and a hydrolyzing alkoxyl group.

[Claim 4] A can according to claim 1, wherein said silane coupling agent-treated layer is a layer treated with a silane containing an organic substituent and a hydrolyzing alkoxyl group and is, then, treated with a silane coupling agent solution comprising an amino group-containing silane solution and/or an epoxy group-containing silane solution.

[Claim 5] A can according to any one of claims 1 to 4, wherein said thermoplastic polyester resin layer is a copolymerized resin film of a polyethylene terephthalate.

[Detailed Description of the Invention]

[0001]

[Technical Field to which the Invention Belongs]

The present invention relates to a can obtained by press-forming a steel plate coated with an organic resin. More specifically, the invention relates a can having a reduced thickness formed by using a surface-treated steel plate without using hexavalent chromium in the step of production and exhibiting excellent work adhesion property and corrosion resistance and is adapted to containing even highly corrosive contents.

[0002]

[Prior Art]

There has heretofore been used a two-piece can having a can wall and a can bottom formed integrally together without a junction portion in the can wall as a metal container for containing foods, beverages and aerosols. The two-piece can is formed through severe workings such as draw-ironing, draw working followed by stretch working, or draw working followed by stretch working and, further, ironing working (stretch-ironing).

The two-piece can has been produced by using an organic resin-coated steel plate obtained by coating a steel plate with an organic resin. From the standpoint of adhesion between the steel blank and the organic resin coating, work adhesion property and corrosion resistance after the working, in particular, an electrolytic chromium/chromium oxide-coated steel plate (tin-free steel, hereinafter referred to as TFS) has been extensively used for producing cans having a reduced thickness through a severe working in which a ratio represented by the following formula (1) and an average plate thickness reduction ratio represented by the following formula (2) are,

$$\text{Height (H) of can/can diameter (D)} \geq 1 \quad \text{--- (1)}$$

$$\{(\text{Initial plate thickness (t}_0\text{)} - \text{can wall thickness (t)})/\text{initial plate thickness (t}_0\text{)}\}$$

$$\times 100 \geq 20\% \quad \text{--- (2)}$$

[0003]

Japanese Unexamined Patent Publication (Kokai) No. 11-140691 teaches that a thermoplastic resin-coated steel plate obtained by forming a silane-treated film and a thermoplastic resin film on the TFS is suited for the use that is to be subjected to a severe working.

[0004]

[Problems to be Solved by the Invention]

The resin-coated steel plate using the above TFS features excellent adhesion of the coated layer even when subjected to the above severe working and exhibits excellent corrosion resistance after the working. However, the TFS is the one obtained through a cathodic electrolytic treatment in a treating solution containing hexavalent chromium followed by the washing with water. Though hexavalent chromium is not contained in the TFS surface-treating film which is the finally formed product, the treating solution contains harmful hexavalent chromium arousing many problems from the environmental point of view.

[0005]

That is, the drain water and exhaust gases of the treating solution containing hexavalent chromium must be completely treated so will not to be drained to the external side. Therefore, a tremendous amount of cost is required for the construction of the drain water treatment facility, exhaust gas treatment facility, and disposal of the waste. Besides, transporting the sludge of waste water and disposal thereof encounter stringent regulations. It has therefore been desired to produce thin cans through the above-mentioned severe working but by using a resin-coated steel plate obtained by using a metal blank other than the TFS.

[0006]

It is, therefore, an object of the present invention to provide a can formed by press-forming a chrome-free steel plate coated with an organic resin featuring excellent film adhesion property, workability and corrosion resistance even when the

thickness is reduced through severe working.

[0007]

[Means for Solving the Problem]

According to the present invention, there is provided a can having excellent corrosion resistance formed by press-forming a pre-coated steel plate which is obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer of an alloy of iron and at least one metal selected from tin, zinc and nickel, containing tin in an amount in a range of not smaller than 0.05 g/m^2 but smaller than 1.5 g/m^2 in the case of a tin-containing alloy, or containing zinc or nickel in an amount in a range of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 in the case of a zinc- or nickel-containing alloy, a silane coupling agent-treated layer containing Si in an amount of 0.8 to 18 mg/m^2 , and a thermoplastic polyester resin layer having a thickness of 8 to $42 \text{ }\mu\text{m}$ in this order from the side of the steel plate.

[0008]

In the press-formed can of the present invention, it is desired that:

- (1) The silane coupling agent-treated layer is a layer formed by the treatment by using an amino group-containing silane solution and/or an epoxy group-containing silane coupling agent solution;
- (2) The silane coupling agent-treated layer is a layer formed by the treatment by using a mixed solution of a silane coupling agent containing an amino group and/or an epoxy group and a silane containing an organic substituent and a hydrolyzing alkoxyl group;
- (3) The silane coupling agent-treated layer is a layer treated with a silane containing an organic substituent and a hydrolyzing alkoxyl group and is, then, treated with a silane coupling agent solution comprising an amino group-containing silane solution and/or an epoxy group-containing silane solution; and

(4) The thermoplastic polyester resin layer is a copolymerized resin film of a polyethylene terephthalate.
[0009]

[Embodiments of the Invention]

According to the present invention, a pre-coated steel plate obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer of iron and at least one metal selected from tin, zinc and nickel, a silane coupling agent-treated layer, and a thermoplastic polyester resin layer, is subjected to severe workings such as draw-ironing, draw working followed by stretch working, or draw working followed by stretch working and, further, ironing working (stretch-ironing) to obtain a press-formed can.

[0010]

As shown in Fig. 1, the pre-coated steel plate of the present invention used for forming a press-formed can is a steel plate coated with an organic resin, comprising a steel plate 1 and, on at least the inner surface side of the steel plate 1, an alloy layer 2, a silane coupling agent-treated layer 3 and a thermoplastic polyester resin layer 4. The pre-coated steel plate is capable of providing a press-formed can that exhibits excellent workability, film adhesion property and excellent corrosion resistance after the working even when the thickness thereof is reduced through a severe working in which a ratio of the height of the can and the can diameter (above formula (1)) and the plate thickness reduction ratio of the can wall (above formula (2)) are,

$$\text{Height of can (H)/can diameter (D)} \geq 1 \quad \text{--- (1)}$$

$$\begin{aligned} &\{(\text{Initial plate thickness (t}_0) - \text{can wall} \\ &\text{thickness (t)})/\text{initial plate thickness (t}_0)\} \\ &\times 100 \geq 20 \quad \text{--- (2)} \end{aligned}$$

or even when it is subjected to a severe working such as flanging or necking.

[0011]

The TFS used as a metal blank of the organic resin-coated

steel plate has a metal chromium layer and a chromium oxide hydrate layer formed on the steel plate exhibiting excellent adhesion to the resin coating and imparting corrosion resistance, resistance against rust and resistance against sulfur staining. According to the present invention, there are formed an alloy layer of iron and at least one metal selected from tin, zinc and nickel in a particular amount, and a silane coupling agent-treated layer on the surface of the steel plate, to withstand severe working, to exhibit excellent adhesiveness to the organic resin coating and to exhibit corrosion resistance and rust resistance.

Namely, upon combining the alloy layer having excellent corrosion resistance and durability with the silane coupling agent-treated layer, it is allowed to improve adhesion of the thermoplastic polyester layer to the alloy layer to impart workability that withstands severe working.

Further, the layer treated with the silane coupling agent itself contributes to improving the durability and resistance against water while suppressing gases from permeating toward the alloy layer and, hence, suppressing the formation of an oxide film of the alloy layer. This prevents the deterioration of adhesion of the thermoplastic polyester resin layer caused by the formation and growth of the oxide film.

[0012]

[Pre-coated steel plate]

The press-formed can of the present invention has an important feature in that it is made of a pre-coated steel plate obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer containing iron and at least one metal selected from tin, zinc and nickel, a silane coupling agent-treated layer containing Si in a particular amount, and a thermoplastic polyester resin layer having a particular thickness.

[0013]

[Alloy layer]

The alloy layer formed on the steel plate on at least the

side that becomes the inner surface of the can contains iron and at least one or more metals selected from tin, zinc and nickel. Formation of the alloy layer on the steel plate improves the corrosion resistance of the steel plate itself, and its combination with the layer treated with the silane coupling agent improves the work adhesion property and improves the corrosion resistance after the working.

In the present invention, it is important that the alloy component constituting the alloy layer together with iron contains tin, zinc or nickel. As will become obvious from Examples appearing later, when there is used cobalt or molybdenum other than the above components as the alloy component (Comparative Examples 7 and 8) or when there is formed no alloy layer (Comparative Example 9), the work adhesion property becomes poor and, particularly, the adhesion becomes poor at the necking portion and the flanging portion where the degree of working is great, developing corrosion at the worked portions and without offering satisfactory corrosion resistance.

[0014]

The alloy layer comprises iron and one or more metals selected from tin, zinc and nickel. In the alloy layer containing tin, it is important that the content of tin is not smaller than 0.05 g/m^2 but is smaller than 1.5 g/m^2 and, particularly, not smaller than 0.1 g/m^2 but is smaller than 1.3 g/m^2 . In the alloy layer containing zinc or nickel, it is desired that the content of zinc or nickel is not smaller than 0.03 g/m^2 but is smaller than 1.8 g/m^2 and, particularly, not smaller than 0.1 g/m^2 but is smaller than 1.2 g/m^2 .

That is, when the contents of metals constituting the alloy layer together with iron do not lie within the above ranges, the work adhesion property becomes poor, and corrosion occurs at the double-seamed portion and at the necking portion, and the corrosion resistance becomes inferior as will become obvious from Examples appearing later (Comparative Examples 1 to 6).

In the case of the alloy layer containing nickel and/or zinc in addition to iron and tin, the alloy layer becomes fine. Therefore, the content of tin may lie within the above range.
[0015]

To form the alloy layer on the surface of the steel plate, for example, to form the tin-iron alloy layer, the steel plate is plated with a predetermined amount of tin followed by heating at a temperature not lower than the melting point of tin and, further, followed by cooling. To form the tin-iron-nickel alloy layer, the steel plate is plated with nickel and, further, with tin followed by heating and cooling. The tin-zinc-iron alloy layer can also be formed in the same manner.

[0016]

[Silane coupling agent-treated layer]

The silane coupling agent-treated layer formed on the alloy layer works to improve the adhesion between the alloy layer and the thermoplastic polyester resin layer owing to the reaction group possessed by the silane coupling agent. Further, the silane coupling agent-treated layer helps improve the durability and resistance against water while suppressing the permeation of gases toward the alloy layer and, hence, suppressing the formation of an oxide film of the alloy layer. This prevents a deterioration in the adhesion to the organic resin coating layer caused by the formation and growth of the oxide film.

It is important that the silane coupling agent-treated layer is so formed that the amount of Si is 0.8 to 18 mg/m² and, particularly, 1 to 15 mg/m². When the amount of Si is smaller than the above range, the work adhesion property is poor and satisfactory corrosion resistance is not obtained (Comparative Example 10). When the amount of Si is greater than the above range, the unreacted silane coupling agent undergoes the self-condensation making it difficult to obtain satisfactory work adhesion property or satisfactory corrosion resistance (Comparative Example 11).

[0017]

The silane coupling agent for forming the silane coupling agent-treated layer possesses a reaction group that chemically bonds to the thermoplastic polyester resin and a reaction group that chemically bonds to the steel plate, and may comprise an organosilane having a reaction group such as amino group, epoxy group, methacryloxy group or mercapto group and a hydrolyzing alkoxyl group such as methoxy group or ethoxy group, or may comprise a silane having an organic substituent such as methyl group, phenyl group, epoxy group or mercapto group and a hydrolyzing alkoxyl group.

Concrete examples of the silane coupling agent that can be preferably used in the present invention include γ -APS (γ -aminopropyltrimethoxysilane), γ -GPS (γ -glycidoxypropyltrimethoxysilane), BTSPA (bistrimethoxysilylpropylaminosilane), and N- β (aminoethyl) γ -aminopropyltrimethoxysilane.
[0018]

To form the silane coupling agent-treated layer on the steel plate, the solution of the above-mentioned silane coupling agent is applied onto the alloy layer, or the steel plate forming the alloy layer is immersed in the solution of the silane coupling agent and, then, an excess of solution is removed by using squeeze rolls. A preferred combination of the silane coupling agent solutions and the order of treatment are as described below.

- (1) A layer is formed by the treatment by using an amino group-containing silane solution and/or an epoxy group-containing silane coupling agent solution.
- (2) A layer is formed by the treatment by using a mixed solution of a silane coupling agent containing an amino group and/or an epoxy group and a silane containing an organic substituent and a hydrolyzing alkoxyl group.
- (3) A layer is formed by the treatment with a silane containing an organic substituent and a hydrolyzing alkoxyl group and, then, with a silane coupling agent solution comprising an amino group-containing silane solution and/or an epoxy

group-containing silane solution.

[0019]

[Thermoplastic polyester resin layer]

In the present invention, the thermoplastic polyester resin layer formed on the silane coupling agent-treated layer to work as a protection film on the surface layer, little adsorbs fragrant components in the content and exhibits excellent barrier property against the corrosive components and excellent shock resistance.

The polyester resin forming the thermoplastic polyester resin layer can be a widely known polyester resin derived from a carboxylic acid component and an alcohol component, and may be a homopolyester, a copolymerized polyester, or a blend of two or more kinds thereof.

[0020]

As the carboxylic acid component, there can be used terephthalic acid, isophthalic acid, naphthalenedicarboxylic acid, *p*- β -oxyethoxybenzoic acid, biphenyl-4,4'-dicarboxylic acid, diphenoxyethane-4,4'-dicarboxylic acid, 5-sodiumsulfoisophthalic acid, hexahydroterephthalic acid, adipic acid, sebacic acid, trimellitic acid, and pyromellitic acid.

As the alcohol component, further, there can be exemplified ethylene glycol, 1,4-butanediol, propylene glycol, neopentyl glycol, 1,6-hexylene glycol, diethylene glycol, triethylene glycol, cyclohexane dimethanol, glycerol, trimethylolpropane, pentaerythritol, dipentaerythritol and sorbitan.

[0021]

Among the known thermoplastic polyester resins according to the present invention, it is particularly desired to use a polyethylene terephthalate type copolymer resin, i.e., an ethylene terephthalate type copolymerized polyester resin in which not less than 50 mol% of the carboxylic acid component is a terephthalic acid, and not less than 50 mol% of the alcohol component is an ethylene glycol component. Desirably, there

can be used a polyethylene terephthalate/isophthalate containing 3 to 18 mol% of isophthalic acid as a carboxylic acid component.

It is desired that the polyester resin that is used has a molecular weight large enough for forming a film and has an inherent viscosity $[\eta]$ as measured in the ortho-chlorophenol at 25°C of 0.6 to 1.2.

[0022]

In the pre-coated steel plate used in the invention, it is important that the thermoplastic polyester resin layer has a thickness in a range of 8 to 42 μm and, particularly, 10 to 40 μm from the standpoint of balance between protecting the steel plate on which the alloy layer is formed and the workability. When the thickness of the thermoplastic polyester resin layer is smaller than the above range, the resin layer loses barrier property due to the reduced thickness, corrosion occurs due to the permeation of the content, cracks occur in the resin layer during the working for reducing the thickness, and the corrosion tends to occur at an increased probability. Further, when the thickness becomes greater than the above range, the resin layer itself exhibits increased rigidity, and the work adhesion property becomes poor in the portions subjected to the severe working such as necking portion or double-seamed portion.

Any known means can be employed, such as extrusion coating method, cast film heat-adhesion method or film heat-adhesion method for forming the thermoplastic polyester resin layer on the steel plate on which there have been formed the alloy layer and the silane coupling agent-treated layer.

When the polyester film is used, the film is obtained by a T-die method or an inflation film-forming method. The film may be an undrawn film formed by the cast-molding method by quickly cooling the extruded film, or may be a biaxially stretched film produced by biaxially drawing the film at a drawing temperature successively or simultaneously and heat-setting the film after it has been drawn.

[0023]

[Steel plate]

The steel plate used in the present invention may be a widely known cold rolled steel plate that has heretofore been used for producing cans, and has a plate thickness of about 0.1 to about 0.4 mm.

[0024]

[Layer constitution of the pre-coated steel plate]

As described above, the pre-coated steel plate used in the present invention has the alloy layer, the silane coupling agent-treated layer and the thermoplastic polyester resin layer that are formed in this order on at least the surface of the steel plate that becomes the inner surface of the can. As required, further, there can be formed other layers. Namely, there can be formed the thermoplastic polyester resin layer on the surface of the steel plate that becomes the outer surface of the can and, besides, a white coated layer and a printed layer may be formed on the thermoplastic polyester resin layer on the outer surface of the can.

[0025]

(Press-formed can)

To press-form a can according to the invention, the above pre-coated steel plate is subjected to the known press forming such as drawing, draw and redrawing, draw and wall ironing, draw and stretching, or draw and stretching and ironing (stretch-ironing) and, further, to the doming, trimming, flanging and necking, to obtain a two-piece can or a one-piece can without seam on the side surface.

The press-formed can of the present invention exhibits particularly excellent effects when its thickness is reduced being subjected to such a severe working that the ratio of height (H) of can/can diameter (D) is in a range of not smaller than 1 and, particularly, 1.1 to 3.0 and that the average plate thickness reduction ratio of the can wall $\{(initial\ plate\ thickness\ (t_0) - can\ wall\ plate\ thickness\ (t))/initial\ plate\ thickness\ (t_0)\} \times 100$ is not smaller than 20% and, particularly,

25 to 70%.

[0026]

In producing the press can of the present invention, the thermoplastic polyester resin layer on the surface imparts a sufficient degree of lubricating property. To further enhance the lubricating property, however, a lubricant such as various oils and fats or waxes may be applied in small amounts to carry out the working on the lubricating solid surface.

[0027]

[EXAMPLES]

The evaluation and the testing were as described below throughout Examples and Comparative Examples.

1. Work adhesion property.

A wax-type lubricant was applied onto both surfaces of the pre-coated steel plate, and a disk of a diameter of 155 mm was punched by press to obtain a shallow drawn cup. The shallow drawn cup was then subjected to the stretch ironing working to obtain a cup having a cup diameter of 66 mm, a cup height of 128 mm and an average plate thickness reduction ratio of the can wall portion of 55%. The cup was subjected to the doming according to an ordinary method, heat-treated at 215°C, and was, then, left to cool, followed by the trimming of the open edge portion, printing on the curved surface, baking and drying, necking and flanging to obtain a can of a reduced thickness having a capacity of 350 g. The can was observed by eyes for its state of adhesion of the metal blank and the resin-coated steel plate on the inner and outer surfaces of the can at the can wall portion, necking portion, and flanging portion, and was evaluated as follows:

○: No delamination.

△: Delamination area is smaller than 1 mm².

X: Delamination area is not smaller than 1 mm².

[0028]

2. Exposure of metal after the can has been formed.

The can after having been formed was filled with an aqueous

solution containing 1% of sodium chloride, and an electric current flowing across the electrode and the can was measured by using an enamel rater and was regarded to be the exposure of a metal.

[0029]

3. Evaluation of testing of the can.

The can that has been formed was filled with 350 g of coke and was double seamed with an aluminum end. The can was stored at 37°C for 6 months.

*Amount of iron dissolved.

The amount of iron in the content after stored and the amount of iron in the content before filled were measured by the atomic absorption method, and a difference therebetween was regarded to be the amount of dissolution of iron. The number of n was 24 cans, and an arithmetic average value of 24 cans was used.

*Inner surface state of the can.

After stored, the content was removed, and the inner surface of the can was washed with water to observe the corroding state on the inner surface of the can and the discolored state by eyes and by using a microscope.

[0030]

(Example 1)

A tin-plated layer was formed on both surfaces of a cold rolled steel plate having a thickness of 0.18 mm and a tempering degree of DR7 by depositing tin in an amount of 0.1 g/m² on each surface, followed by a reflow treatment to transform all metal tin into an iron-tin alloy layer. The steel plate was then immersed in an aqueous solution containing 3% of γ -APS (γ -aminopropyltrimethoxysilane), and an excess of the γ -APS solution was readily squeezed off by using squeeze rolls to obtain a surface-treated steel plate having a silane coupling agent-treated layer containing Si in an amount of 5 mg/m². Next, the surface-treated steel plate was heated at a temperature higher than a melting point of the film by 10°C to heat-laminate a copolymerized polyester (m.p., 228°C) cast film comprising

a copolymerized polyester (terephthalic acid/isophthalic acid (weight ratio of 88/12) and an ethylene glycol maintaining a thickness of 20 μm on both surfaces at a laminate roll temperature of 150°C and a passing speed of 150 m/min., immediately followed by cooling with water to obtain a pre-coated steel plate. A wax-type lubricant was applied onto both surfaces of the pre-coated steel plate, and a disk of a diameter of 155 mm was punched by press to obtain a shallow drawn cup. Next, the shallow drawn cup was subjected to the stretch ironing to obtain a stretch-ironed cup having a cup diameter of 66 mm, a cup height of 128 mm and an average plate thickness reduction ratio of the can wall of 55%. The cup was subjected to the doming according to an ordinary method, heat-treated at 215°C, and was, then, left to cool, followed by the trimming of the open edge portion, printing on the curved surface, baking and drying, necking and flanging to obtain a can of a reduced thickness having a capacity of 350 g. Next, the can was filled with coke, and was double seamed with an end. After stored, the inner surface state of the can was examined.

Table 1 shows compositions of the alloy layers of the pre-coated steel plates, amounts of tin and other metals in the alloy layers, kinds of the surface treatment, thickness of treatment, kinds and thickness of organic coating materials, and Table 2 shows the evaluated results of the test of the cans.

[0031]

(Examples 2 to 6, Comparative Examples 1 and 2).

Pre-coated members were prepared and cans were produced in the same manner as in Example 1 but varying the amount of tin in the alloy layers as represented by the amounts in Table 1, to evaluate them. The results were as shown in Table 2.

[0032]

(Example 7)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but plating nickel in an amount of 0.3 g/m² on each surface prior to plating tin, and plating tin in an amount of 0.6 g/m² on each surface to evaluate them.

The results were as shown in Table 2.

[0033]

(Example 8)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but plating zinc in an amount of 0.3 g/m^2 on each surface prior to plating tin, and plating tin in an amount of 0.6 g/m^2 on each surface to evaluate them. The results were as shown in Table 2.

[0034]

(Examples 9 to 13, Comparative Examples 3 and 4)

Pre-coated members were prepared and cans were produced in the same manner as in Example 1 but plating an iron-nickel alloy layer containing nickel in amounts as shown in Table 1 instead of plating tin, to evaluate them. The results were as shown in Table 2.

[0035]

(Examples 14 to 18, Comparative Examples 5 and 6)

Pre-coated members were prepared and cans were produced in the same manner as in Example 1 but plating zinc in amounts as shown in Table 1 instead of plating tin, and plating an iron-zinc alloy layer by the reflow treatment, to evaluate them. The results were as shown in Table 2.

[0036]

(Comparative Example 7)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but plating an iron-cobalt alloy containing cobalt in an amount of 0.8 mg/m^2 instead of plating tin, to evaluate them. The results were as shown in Table 2.

[0037]

(Comparative Example 8)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but plating an iron-molybdenum alloy containing molybdenum in an amount of 0.8 mg/m^2 instead of plating tin, to evaluate them. The results were as shown in Table 2.

[0038]

(Comparative Example 9)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but plating a tin layer on both surfaces by depositing tin in an amount of 0.1 g/m^2 on each surface, and omitting the reflow treatment so as not to form the iron-tin alloy layer, to evaluate them. The results were as shown in Table 2.

[0039]

(Examples 19 to 21, Comparative Examples 10 and 11)

Pre-coated members were prepared and cans were produced in the same manner as in Example 1 but selecting the amount of tin in the alloy layers to be 0.6 g/m^2 and selecting the thickness of the surface treatment to be as shown in Table 1 in terms of the Si amount, to evaluate them. The results were as shown in Table 2.

[0040]

(Example 22)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with a 3% of γ -GPS (γ -glycidoxypropyltrimethoxysilane) water ethanol solution instead of using a γ -APS aqueous solution and forming a film by the treatment containing Si in an amount of 5 mg/m^2 , to evaluate them. The results were as shown in Table 2.

[0041]

(Example 23)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with a 3% of BTSE (bis-1,2-(triethoxysilyl)ethane) water ethanol solution instead of using a γ -APS aqueous solution, followed by the treatment with a 3% of γ -APS aqueous solution, and forming a film by the treatment containing Si in a total amount of 10 mg/m^2 , to evaluate them. The results were as shown in Table 2.

[0042]

(Example 24)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with a water ethanol solution of a mixture of 3% of BTSPS (bistrimethoxysilylpropyltetra sulfide) and 3% of γ -APS instead of using a γ -APS aqueous solution, and forming a film by the treatment containing Si in an amount of 10 mg/m², to evaluate them. The results were as shown in Table 2.

[0043]

(Comparative Example 12)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with a 3% tetraethoxysilane solution instead of using a γ -APS aqueous solution and forming a film by the treatment containing Si in an amount of 5 mg/m², to evaluate them. The results were as shown in Table 2.

[0044]

(Comparative Example 13)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with a 3% BTSE (bis-1,2-(triethoxysilyl)ethane) water ethanol solution instead of using a γ -APS aqueous solution, and forming a film by the treatment containing Si in an amount of 5 mg/m², to evaluate them. The results were as shown in Table 2.

[0045]

(Comparative Example 14)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but subjecting the surface on the tin-iron alloy layer to the electrolytic phosphoric acid treatment instead of the treatment with the γ -APS aqueous solution and forming a film by the treatment containing P in an amount of 2.5 mg/m², to evaluate them. The results were as shown in Table 2.

[0046]

(Comparative Example 15)

A pre-coated member was prepared and a can was produced in the same manner as in Example 1 but treating the surface on the tin-iron alloy layer with tin phosphate instead of using a γ -APS aqueous solution, and forming a film by the treatment containing P in an amount of 2.5 mg/m^2 and Sn in an amount of 2.5 mg/m^2 , to evaluate them. The results were as shown in Table 2.

[0047]

(Examples 25 and 26, Comparative Examples 16 and 17)

Pre-coated members were prepared and cans were produced in the same manner as in Example 1 but using an N- β (aminoethyl) γ -aminopropyltrimethoxysilane as the silane coupling agent, forming a film by the treatment containing Si in an amount of 7 mg/m^2 , and selecting the thickness of the copolymerized polyester film which is an organic coating material as shown in Table 1, to evaluate them. The results were as shown in Table 2.

[0048]

(Examples 27 and 28)

Pre-coated members were prepared and cans were produced in the same manner as in Example 25 but changing the kinds and thickness of the polyester film which is the organic coating material as shown in Table 1, to evaluate them. The results were as shown in Table 2.

[0049]

(Comparative Example 18)

A pre-coated member was prepared and a can was produced in the same manner as in Example 25 but using a polypropylene film of a thickness of $25 \text{ }\mu\text{m}$ as the organic coating material instead of using the copolymerized polyester film, and laminating the films by using an urethane-type adhesive, to evaluate them. The results were as shown in Table 2.

[0050]

(Comparative Example 19)

A pre-coated member was prepared and a can was produced

in the same manner as in Example 25 but using a polyethylene film of a thickness of 25 μm as the organic coating material instead of using the copolmerized polyester film, and laminating the films by using an urethane-type adhesive, to evaluate them. The results were as shown in Table 2.

[0051]

(Comparative Example 20)

A pre-coated member was prepared and a can was produced in the same manner as in Example 25 but using an epoxyacrylic coating material which is an organic coating material instead of using the copolymerized polyester film, roll-coating the coating material in such an amount that the thickness after baking was 10 μm and baking the coating material at 200°C for 10 minutes, to evaluate them. The results were as shown in Table 2.

[0052]

(Comparative Example 21)

A pre-coated member was prepared and a can was produced in the same manner as in Example 25 but using an epoxyphenol-type coating material which is an organic coating material instead of using the copolymerized polyester film, roll-coating the coating material in such an amount that the thickness after baking was 10 μm and baking the coating material at 200°C for 10 minutes, to evaluate them. The results were as shown in Table 2.

[0053]

(Comparative Example 22)

A pre-coated member was prepared and a can was produced in the same manner as in Example 25 but using a vinylorganosol-type coating material which is an organic coating material instead of using the copolymerized polyester film, roll-coating the coating material in such an amount that the thickness after baking was 15 μm and baking the coating material at 200°C for 10 minutes, to evaluate them. The results were as shown in Table 2.

[0054]

Table 1

Sample	Alloy layer		Surface treatment		Organic coating material	
	Composition	Amount of metal (g/m ²)	Kind	Treating thickness (mg/m ²)	Kind	Thickness (μm)
Ex. 1	Sn-Fe	Sn 0.1	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 2	Sn-Fe	Sn 0.3	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 3	Sn-Fe	Sn 0.5	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 4	Sn-Fe	Sn 0.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 5	Sn-Fe	Sn 1.0	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 6	Sn-Fe	Sn 1.3	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 1	Sn-Fe	Sn 0.05	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 2	Sn-Fe	Sn 1.5	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 7	Sn-Fe-Ni	Sn 0.6	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 8	Sn-Fe-Zn	Sn 0.6	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 9	Fe-Ni	Ni 0.08	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 10	Fe-Ni	Ni 0.1	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 11	Fe-Ni	Ni 0.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 12	Fe-Ni	Ni 1.2	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 13	Fe-Ni	Ni 1.5	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 3	Fe-Ni	Ni 0.03	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 4	Fe-Ni	Ni 1.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 14	Fe-Zn	Zn 0.08	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 15	Fe-Zn	Zn 0.1	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 16	Fe-Zn	Zn 0.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 17	Fe-Zn	Zn 1.2	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 18	Fe-Zn	Zn 1.5	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 5	Fe-Zn	Zn 0.03	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 6	Fe-Zn	Zn 1.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 7	Fe-Co	Co 0.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 8	Fe-Mo	Mo 0.8	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 9	-	-	γ-APS ¹⁾	5	PET/IA (12%) ⁵⁾	20
Ex. 19	Sn-Fe	Sn 0.6	γ-APS ¹⁾	1	PET/IA (12%) ⁵⁾	20
Ex. 20	Sn-Fe	Sn 0.6	γ-APS ¹⁾	3	PET/IA (12%) ⁵⁾	20

1) γ-APS: silane coupling agent, γ-aminopropyltrimethoxysilane

5) PET/IA (12%): polyethylene terephthalate/isophthalate (copolymerizing ratio, 12%)

Table 1 (continued)

Sample	Alloy layer		Surface treatment		Organic coating material	
	Composition	Amount of metal (g/m ²)	Kind	Treating thickness (mg/m ²)	Kind	Thickness (μm)
Ex. 21	Sn-Fe	Sn 0.6	γ-APS ¹⁾	Si, 15	PET/IA (12%) ⁵⁾	20
Comp. Ex. 10	Sn-Fe	Sn 0.6	γ-APS ¹⁾	Si, 0.5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 11	Sn-Fe	Sn 0.6	γ-APS ¹⁾	Si, 20	PET/IA (12%) ⁵⁾	20
Ex. 22	Sn-Fe	Sn 0.6	γ-GPS ²⁾	Si, 5	PET/IA (12%) ⁵⁾	20
Ex. 23	Sn-Fe	Sn 0.6	BTSE ³⁾ → γ-APS 2-step treatment	Si, 10	PET/IA (12%) ⁵⁾	20
Ex. 24	Sn-Fe	Sn 0.6	mixed solution of BTSPS, γ-APS	Si, 10	PET/IA (12%) ⁵⁾	20
Comp. Ex. 12	Sn-Fe	Sn 0.6	tetraethoxysilane	Si, 5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 13	Sn-Fe	Sn 0.6	BTSE ³⁾	Si, 5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 14	Sn-Fe	Sn 0.6	phosphoric acid	P, 2.5	PET/IA (12%) ⁵⁾	20
Comp. Ex. 15	Sn-Fe	Sn 0.6	tin phosphate	P, 2.5	PET/IA (12%) ⁵⁾	20
Ex. 25	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Sn, 2.5	PET/IA (12%) ⁵⁾	10
Ex. 26	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PET/IA (12%) ⁵⁾	40
Comp. Ex. 16	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PET/IA (12%) ⁵⁾	7
Comp. Ex. 17	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PET/IA (12%) ⁵⁾	45
Ex. 27	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PET/IA (12%) ⁵⁾	25
Ex. 28	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PET/IA (8%) ⁶⁾	25
Comp. Ex. 18	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	homo PET	25
Comp. Ex. 19	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PP	25
Comp. Ex. 20	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	PE	25
Comp. Ex. 21	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	epoxyacrylic paint	10
Comp. Ex. 22	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	epoxyphenol paint	10
Comp. Ex. 22	Sn-Fe	Sn 0.8	N-β (aminoethyl) γ-APS ⁷⁾	Si, 7	vinylorganosol	15

1) γ-APS: silane coupling agent, γ-aminopropyltrimethoxysilane

2) γ-GPS: silane coupling agent, γ-glycidoxypropyltrimethoxysilane

3) BTSE: silane bis-1,2-(triethoxysilyl)ethane

4) BTSPS: silane bistrimethoxysilylpropyltetrasulfide

5) PET/IA (12%): polyethylene terephthalate/isophthalate (copolymerizing ratio, 12%)

6) PET/IA (8%): polyethylene terephthalate/isophthalate (copolymerizing ratio, 8%)

7) N-β (aminoethyl) γ-APS: silane coupling agent N-β (aminoethyl) γ-aminopropyltrimethoxysilane

Table 2

Experiment	Work adhesion		Exposure of metal (mA)	Dissolved iron (ppm)	Can testing	
	Can wall	Necking portion			Inner surface of can	
Ex. 1	○	○	0.00	0.00	normal	
Ex. 2	○	○	0.00	0.00	normal	
Ex. 3	○	○	0.00	0.00	normal	
Ex. 4	○	○	0.00	0.00	normal	
Ex. 5	○	○	0.00	0.00	normal	
Ex. 6	○	○	0.00	0.00	normal	
Comp. Ex. 1	○	○	0.10	2.02	double-seamed portion partly corroded	
Comp. Ex. 2	○	△	2.50	5.31	necking portion, double-seamed portion corroded	
Ex. 7	○	○	0.00	0.00	normal	
Ex. 8	○	○	0.00	0.00	normal	
Ex. 9	○	○	0.00	0.00	partly discolored under the film	
Ex. 10	○	○	0.00	0.00	normal	
Ex. 11	○	○	0.00	0.00	normal	
Ex. 12	○	○	0.00	0.00	normal	
Ex. 13	○	○	0.00	0.00	partly discolored under the film	
Comp. Ex. 3	○	○	0.11	2.21	necking portion, double-seamed portion corroded	
Comp. Ex. 4	○	△	2.52	5.95	necking portion, double-seamed portion corroded	
Ex. 14	○	○	0.00	0.00	partly discolored under the film	
Ex. 15	○	○	0.00	0.00	normal	
Ex. 16	○	○	0.00	0.00	normal	
Ex. 17	○	○	0.00	0.00	normal	
Ex. 18	○	○	0.00	0.00	partly discolored under the film	
Comp. Ex. 5	○	○	0.13	3.00	necking portion, double-seamed portion corroded	
Comp. Ex. 6	○	△	2.55	6.02	necking portion, double-seamed portion corroded	
Comp. Ex. 7	○	△	0.56	2.45	necking portion, double-seamed portion partly corroded	

Table 2 (continued)

Experiment	Work adhesion		Exposure of metal (mA)	Can testing	
	Can wall	Necking portion	Flanging portion	Dissolved iron (ppm)	Inner surface of can
Comp. Ex. 8	O	Δ	Δ	3.13	necking portion, double-seamed portion partly corroded
Comp. Ex. 9	O	O	Δ	0.92	double-seamed portion partly corroded
Ex. 19	O	O	O	0.00	normal
Ex. 20	O	O	O	0.00	normal
Ex. 21	O	O	O	0.00	normal
Comp. Ex. 10	O	Δ	×	7.23	necking portion, double-seamed portion corroded
Comp. Ex. 11	O	Δ	×	8.54	necking portion, double-seamed portion corroded
Ex. 22	O	O	O	0.00	normal
Ex. 23	O	O	O	0.00	normal
Ex. 24	O	O	O	0.00	normal
Comp. Ex. 12	Δ	Δ	×	8.17	necking portion, double-seamed portion corroded
Comp. Ex. 13	Δ	Δ	×	8.31	necking portion, double-seamed portion corroded
Comp. Ex. 14	Δ	×	×	11.2	inner surface as a whole corroded
Comp. Ex. 15	Δ	×	×	10.5	inner surface as a whole corroded
Ex. 25	O	O	O	0.00	normal
Ex. 26	O	O	O	0.00	normal
Comp. Ex. 16	O	O	O	1.20	partly corroded under the film
Comp. Ex. 17	O	Δ	Δ	2.77	necking portion, double-seamed portion corroded
Ex. 27	O	O	O	0.00	normal
Ex. 28	O	O	O	0.00	normal
Comp. Ex. 18	O	O	Δ	1.82	double-seamed portion corroded
Comp. Ex. 19	O	O	Δ	2.12	double-seamed portion corroded
Comp. Ex. 20	O	Δ	Δ	29.3	inner surface as a whole corroded
Comp. Ex. 21	O	Δ	Δ	25.7	inner surface as a whole corroded
Comp. Ex. 22	O	Δ	Δ	20.2	inner surface as a whole corroded

[0056]

The following will be learned from the above Examples and Comparative Examples.

Examples 1 to 18 and Comparative Examples 1 to 9 have tested stretch-ironed cans having a reduced thickness made of pre-coated steel plates obtained by varying the composition and amounts of components of the alloy layer formed on the steel plate. It will be learned that the cans made of a pre-coated steel plate provided with an alloy layer containing iron and at least one kind of metal selected from tin, zinc and nickel, exhibit superior corrosion resistance to those of cans made of pre-coated steel plates provided with other alloy layers. In particular, excellent corrosion resistance and work adhesion property are exhibited when the steel plate is provided with an alloy layer containing tin in an amount of not smaller than 0.05 g/m^2 but smaller than 1.5 g/m^2 , an iron-tin-nickel alloy layer, an iron-tin-zinc alloy layer, an iron-nickel alloy layer containing nickel in an amount of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 , and with an iron-zinc alloy layer containing zinc in an amount of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 .

[0057]

Examples 19 to 21 and Comparative Examples 10 and 11 have tested stretch-ironed cans having a reduced thickness made of pre-coated steel plates obtained by varying the thickness of the silane coupling agent-treated layer. It will be learned that excellent corrosion resistance and work adhesion property are obtained when the cans are made of pre-coated steel plates having a film of a thickness of an Si amount of 0.8 to 18 mg/m^2 .

[0058]

Examples 22 to 24 and Comparative Examples 12 to 15 have tested deep drawn cans having a reduced thickness made of pre-coated steel plates obtained by using different kinds of surface treating agents. It will be learned that the cans made of pre-coated steel plates obtained by using an organosilane coupling agent having a reaction group such as amino group or

epoxy group and a hydrolyzing alkoxyl group, exhibit superior corrosion resistance and work adhesion property to those of the cans made of pre-coated steel plates obtained by using a silane treating agent without containing such groups, by using a phosphoric acid treating agent, or by using a tin phosphate treating agent.

[0059]

Examples 25 and 26 and Comparative Examples 16 and 17 have tested deep drawn cans having a reduced thickness made of pre-coated steel plates obtained by varying the thickness of the copolymerized polyester film. It will be learned that the cans made of a pre-coated steel plate coated with a polyester film of a thickness of 8 to 24 μm exhibit superior corrosion resistance and work adhesion property to those of the cans made of pre-coated steel plates coated with the polyester film having a thickness without lying in the above range.

[0060]

Examples 27 and 28 and Comparative Examples 18 to 22 have tested the stretch-ironed cans having a reduced thickness made of pre-coated steel plates obtained by using organic coating materials of different kinds. It will be learned that the cans made of a pre-coated steel plate obtained by using an organic coating material of polyester exhibit superior corrosion resistance and work adhesion property to the cans made of pre-coated steel plates obtained by using films and coating materials of other kinds.

[0061]

[Effect of the Invention]

A press-formed can of the present invention is produced by using a chrome-free pre-coated steel plate which is obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer of an alloy of iron and at least one metal selected from tin, zinc and nickel, containing tin in an amount in a range of not smaller than 0.05 g/m^2 but smaller than 1.5 g/m^2 in the case of a tin-containing alloy, or containing zinc or nickel in an

amount in a range of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 in the case of a zinc- or nickel-containing alloy, a silane coupling agent-treated layer containing Si in an amount of 0.8 to 18 mg/m^2 , and a thermoplastic polyester resin layer having a thickness of 8 to $42 \text{ }\mu\text{m}$ in this order from the side of the steel plate. Therefore, there is produced a can featuring excellent work adhesion property of the film as well as excellent workability and corrosion resistance even when the thickness is reduced through a severe working such as draw-ironing, redrawing for reducing the thickness, or stretch-ironing even in those portions that are subjected to a severe working such as flanging and necking.

[Brief Description of the Drawing]

[Fig. 1] is a sectional view illustrating a pre-coated steel plate used for a press-formed can of the present invention.

[Designation of Document] Abstract

[Abstract]

[Problem] To provide a can obtained by press-forming a chrome-free steel plate coated with an organic resin featuring excellent film adhesion property, workability and corrosion resistance even when the thickness is reduced through severe working.

[Means for Solution]

A can having excellent corrosion resistance formed by press-forming a pre-coated steel plate which is obtained by providing, on the surface of the steel plate on at least the side that becomes the inner surface of the can, an alloy layer of an alloy of iron and at least one metal selected from tin, zinc and nickel, containing tin in an amount in a range of not smaller than 0.05 g/m^2 but smaller than 1.5 g/m^2 in the case of a tin-containing alloy, or containing zinc or nickel in an amount in a range of not smaller than 0.03 g/m^2 but smaller than 1.8 g/m^2 in the case of a zinc- or nickel-containing alloy, a silane coupling agent-treated layer containing Si in an amount of 0.8 to 18 mg/m^2 , and a thermoplastic polyester resin layer having a thickness of 8 to $42 \text{ }\mu\text{m}$ in this order from the side of the steel plate.

[Selected Drawing] None